PREPARATION OF TiO_2-ZrO_2-Y_2O_3 COMPOSITE FOR LOAD BEARING IMPLANTS

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BY

PRAKASH RANJAN ROUT

109BM0354

Under the Guidance of

Dr. AMIT BISWAS

NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA, ORISSA-769008

DEPARTMENT OF BIOTECHNOLOGY & MEDICAL ENGINEERING

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DEPARTMENT OF BIOTECHNOLOGY & MEDICAL ENGINEERING

National Institute of Technology,

Rourkela-769008

CERTIFICATE

This is to certify that the report entitled **"PREPARATION OF TiO_2-ZrO_2-Y_2O_3 COMPOSITE FOR LOAD BEARING IMPLANTS"** being submitted by **PRAKASH RANJAN ROUT** towards the fulfillment of the requirement for the degree of Bachelors of Technology in Biomedical Engineering at Department of Biotechnology & Medical Engineering Engineering, NIT Rourkela is a record of bonafide work carried out by him under my guidance and supervision**.**

Dr. AMIT BISWAS

Dept. of Biotechnology and Medical Engg.,

National Institute of Technology,Rourkela

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Thanking you,

PRAKASH RANJAN ROUT

109BM0354

Dept. of Biotechnology and Medical Engg.,

National Institute of Technology, Rourkela

ABSTRACT

The present work deals with the preparation of ceramic composites that can be employed for the development of load bearing bone implants in total knee and hip replacement. Zirconia (ZrO_2) is a bio-inert, strong, and resistant ceramic, while titania (TiO_2) is bioactive but is believed to have poor mechanical properties. It is expected that yttria stabilized ZrO_2-TiO_2 ceramic composites incorporate the desirable properties of both ceramics, so that this composite would exhibit better in-vivo biological properties. The composites of Ti-ZrO_2 (0.2 mass fraction) stabilised with 3 mol% yttria with different particle size were prepared using powder metallurgy. The obtained powders were then sintered at 1300 °C for 1 hr in normal atmosphere subsequent to dry pressing at 130MPa. Sintering led to the oxidation of titanium into titanium oxide(titania).Phase characterization was analyzed by XRD, morphological features were assessed by SEM, hardness was measured by micro-hardness tester and MTT assay was used for the measurement of cell viability in vitro to understand the compatibility of implant in body environment.

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CHAPTER 1

INTRODUCTION

OBJECTIVE

SPECIFIC OBJECTIVE

1.1 INTRODUCTION

The modern day medicine is in a transition from curing damaged/diseased organs of the patient by hectic and time consuming surgical operations to replacing the damaged/diseased organ completely with in vitro synthesized implants. Hard tissue and bone replacements are synthesized mainly from materials having similar chemical, mechanical properties and phase structure to those of the hard tissues. Study on the synthesis of new biomaterials involves the use of the existing biomaterials with a new composite material with improved properties, modification of the microstructure of the present biomaterials and chemical synthesis to form a new novel biomaterial. Metals such as stainless steel and titanium alloy and ceramics such as alumina, and zirconia are common in a variety of implant materials. However, these materials are considerably stiffer than human bone. The elasticity or modulus mismatch between an implant material and the host tissue can cause bone to yield at the implant-bone interface, which leads to implant instability and subsequent failure [1]. A durable bone replacement requires the establishment of a stable bone-implant interface, which requires the careful matching of the mechanical behaviour as well as physico-chemical properties of synthetic implant materials with the natural tissue [2]. Material researchers are currently working on the development of biocompatible materials which can mimic the properties of natural bone. Molecularly designed composite materials that mimic the structure and properties of bone offer the best solution to the problems of interfacial stability and stress shielding of load-bearing prostheses.

As compared to metals, ceramics often cause reduced osteolysis and are regarded as favorable materials for joints or joint surface materials. Titanium and its alloys are the most biocompatible because of their resistance and tolerance to body fluids. It has high

fatigue strength, low weight, & outstanding corrosion resistance. However they posses poor shear strength and wear resistance. Titania (TiO_2) is a ceramic material, which is generally used as a coating material in the femoral head in hip prosthesis. Fine grained TiO_2 particles with a narrow size distribution are desirable for producing advanced ceramics with enhanced reliability [3]. Recently, TiO_2 fine-grained particles have become a topic of extensive biological and physico-chemical investigations. TiO_2 surface layers have been coated on metal substrates and have shown extraordinary biocompatibility. The titania coating has shown promising bioactivity; & has the ability to form chemical bonding with bone in the body. Despite its poor mechanical properties, and being inferior to hydroxyapatite in terms of bone apposition rate and bone bonding strength, titania is relatively stable in a biological environment and is able to provide long-term bone bonding. TiO_2 ceramics are promising for implant applications. Zirconia has high thermal stability, high bending strength, high fracture toughness, high ionic conductivity, good wear resistance. Zirconia can be stabilised with various additives such as CaO, Y_2O_3,MgO etc among which yttria and cerium are the most effective stabilisers. In particular, yttria-stabilised zirconia (ZrO_2), is known to be both hard and tough at room temperature because its addition reduces the temperature of tetragonal to monoclinic transformation of zirconia polymorph. Zirconia ceramics have an advantage over alumina ceramics i.e. they have higher toughness & higher flexural strength & lower youngs modulus.

1.2 OBJECTIVE

The objective of this present study is the preparation of TiO_2 and Yttria stabilized ZrO_2 (3Y-PSZ) composite and to find out their potential application in load bearing implants. In this present work the following specific objectives has to be achieved:-

(i) Selection of appropriate concentration of Ti and Yttria stabilized zirconia for preparing the composite by ball milling.

(ii) Transformation of Titanium to Titania followed by the formation of Titania-Yttria-zirconia composite by sintering at oxidizing atmosphere and to find out the correlation between the microstructure before and after sintering.

(iii) Phase analysis of the samples will be carried out by XRD study before and after sintering to find out any abnormal change.

(iv) The micro hardness test will be carried out to evaluate the hardness of the composite.

(v) Assessment of cell viability of the composite material will be performed by MTT assay.

CHAPTER 2

LITERATURE REVIEW

BIOMATERIALS

BIOCERAMICS

COMPOSITES

TITANIA AND 3Y- PSZ

2.1 BIOMATERIALS

A biomaterial is a nonviable material used in medical device, intended to interact with biological system[4].The most requisite property of all the biomaterials is biocompatibility or non-toxicity which is the definition of a material that is not recognized by the body as a potentially harmful foreign substance and does not produce potentially toxic products in the body environment. Once an implant is placed inside the body, an injury response is initiated by the tissue that results in inflammation as a reaction to any local injury. Not all biocompatible materials are inert in the body but the highly bioactive ones incorporate to the actions of body like providing a host matrix for tissue growth or being slowly replaced by the growing tissue on or near its surface. Biocompatible materials can classified as bioinert, resorbable and bioactive according to tissue response. Formation of a fibrous tissue of variable thickness is induced in bioinert materials, bond at the interfaces is developed on bioactive materials, and resorbable materials are replaced by the surrounding tissue [4]. Mechanical property correlation of the biomaterial to the host or replaced tissue is another important property. Especially in hard tissue replacements in load bearing implants the biomaterial is required to support or share a portion of the load. Wear resistance, Compressive strength, fracture toughness and hardness of biomaterials are important in these cases. The list of biomaterials used in body implants with their properties has been enumerated in Table 2.1[5].

Table. 2.1 list of biomaterials used in body implants with their properties

Type	Properties	Applications
Polymers		
PMMA	High toughness, stability	Bone cement
	Excellent light transmittance	Intraocular lenses
PHEMA	Excellent light transmittance	Soft contact lenses
Polyacrylic acid	High adhesive strength	Dental cement
		Mucosal drug delivery
Polyethylene	High toughness, wear resistance	Catheters
		Acetabular cups in artificial hips
Polypropylene	High tensile strength, chemical	Sutures
	Resistance	Hernia repair
PTFE	High hydrophobicity	Catheters
	Excellent lubricity	Vascular grafts
Poly(dimethyl siloxane)	Low Tg	Finger joints
	Low mechanical properties	Heart valves
	High flexibility	Breast implants
PET	High tensile strength	Arterial grafts
		Fixation of implants
Polyurethane	High fatigue resistance	Pacemaker insulation
		Vascular grafts
Metals		
316L Stainless steel	High corrosion resistance	Bone screws

	High strength	Hip prostheses
Co-Cr-Mo F75	High corrosion resistance Low fatigue resistance	Heart valve stents
Ti-6Al-4V	High strength High corrosion resistance	Heart housing Heart valve stents
Ceramics		
Al_2O_3	High corrosion resistance High wear resistance	Hip prostheses Dental implants
ZrO_2	Low modulus of elasticity High strength	Articulating ball in total hip prosthesis
Hydroxyapatite	High bioactivity Low strength	Dental implants Alveolar ridge Periodontal pocket
Calcium phosphates	High bioactivity	Degradable bone filler
Pyrolytic carbon	High Strength High wear resistance	Heart valves Dental implants
Composites		
Hydroxyapatite-Polyethylene	High bioactivity High toughness	Bone implant
Alumina-Zirconia	High wear resistance	Femoral heads
Natural Materials		
Chitosans		Wound dressing
Collagen		Soft tissue coatings
Gelatin		Artificial heart

2.2 BIOCERAMICS

Ceramics are a versatile group of materials that are abundant in type. Oxides are the most commonly used ceramics also found in great quantities in nature. The chemical synthesis, fabrication and heat treatment processes of oxides are relatively easier and simpler compared to other materials. The desired characteristic properties common to all ceramics are chemical stability, low density, high hardness, low tensile strength and high compressive strength. Ceramics are ideal materials for mobile load bearing components in aggressive environments such as those in automobile engine blocks, refractories and hard tissue replacements. Following contact with body tissue, exposed surface of a biomaterial is covered rapidly with proteins that are adsorbed from the surrounding body fluids. The nature of the adherent protein layer is controlled by the substrate chemistry, due to its effect on wettability and surface charge. Although cells are able to adhere, spread and grow on bare biomaterial surfaces in vitro, proteins adsorbed from the adjacent tissue environment and adherent cells enhance cell attachment, migration and growth. The chemical nature of a biomaterial placed in the body as an implant therefore is important in functioning of the body. Some ceramics that have been tested in vivo do not cause increased activity of immune system when dissolved in body fluid or in contact with body tissues. Such ceramics, mainly oxides, are termed as bioceramics. Bioceramics have the advantage of being compatible with the human body environment. Due to their excellent tribological properties and with their improved fracture toughness and reliability, structural ceramics such as polycrystalline alumina and toughened zirconia have been used as hard tissue replacement materials. One remarkable success of bioceramics as implant materials over the last two decades has been the emergence and clinical use of bioactive ceramics that elicit a specific biological response at the interface of the material resulting in the formation of a strong bond

between the tissue and the material. These bioceramics include calcium phosphates with hydroxyapatite, Bioglasss, A-W glass–ceramic, and other bioactive glasses and glass–ceramics [6]. However, the brittle nature of ceramics such as alumina and the low strength of bioactive ceramics such as hydroxyapatite have limited their scope of clinical applications. Bioceramics can be classified into four groups based on their type of attachment to the surrounding tissues. Dense and nearly inert ceramics get attached to the bone by morphological fixation, or growth of bone into surface irregularities. Porous and inert ceramics are attached to the bone by on-growth of the tissue or biological fixation. Dense and surface reactive ceramics attach directly by chemical bonding to the bone or via bioactive fixation. Resorbable ceramics get attached to the bone by any of the above mechanisms and are slowly replaced by bone.

2.3 COMPOSITE:

Composites are derived materials obtained by combining advantageous properties of metallic/ceramic/polymeric materials to achieve property which is higher than sum total of individual phase characteristics. Three distinct composite classes- Metal-Metal Composite, Polymer-Metal Composite and Ceramic-Metal Composite. It is essential that each component of the composite be biocompatible to avoid degradation between interfaces of the constituents. It has been known that interfaces in these material systems, especially composites play a critical role in observed mechanical properties much like the role of load transfer mechanisms at HAP–collagen interfaces in real bone [7].

2.4. TITANIA AND YTTRIA STABILISED ZIRCONIA:

Pure titania is subject to anatase to rutile phase transformation, although the transformation temperature varies. It was reported that the anatase to rutile phase transformation could be reduced by a small amount of zirconia, which was present in the form of solid solution with the

host titania [8]. With zirconia as the dopant, anatase phase with high crystallinity and high phase stability was maintained even after annealing at 1000^0C for 1 h [9].It has been observed that TiO_2 particles fuse together with increasing sintering temperature,and all of the sintered TiO_2 membranes are porous.In addition the TiO_2 membranes obtained in air are seen to be more compact than that obtained in argon at same temperature.[10]. It has been found that the grain boundary diffusion is faster by about 5 orders of magnitude than the bulk diffusion of titanium in yttria satbilised zirconia.[11].

Zirconia is an inert ceramic in its pure form which possesses extraordinary properties when doped with certain stabilizing oxides such as yttria, magnesia and calcia. It is a well-known polymorph that occurs in three forms: monoclinic (M), tetragonal (T), and cubic (C). Pure zirconia is monoclinic at room temperature. This phase is stable up to 1170℃. Above this temperature it transforms into tetragonal and then into cubic phase at 2370℃. During cooling, a T to M transformation takes place in a temperature range of about 100℃ below 1070℃. The improved mechanical property due to transformation toughening of tetragonal zirconia is utilized in biocomposites as well as conventional ceramic to have a better mechanical property. Obtaining stable sintered zirconia ceramic products is difficult because of the large volume change accompanying the transition from tetragonal to monoclinic (about 9%). Stabilization of the tetragonal polymorph of zirconia over wider range of temperatures is accomplished by substitution of some of the Zr^{4+} ions (ionic radius of 0.82 Å, too small for ideal lattice of fluorite characteristic for the tetragonal zirconia) in the crystal lattice with slightly larger ions, e.g., those of Y^{3+} (ionic radius of 0.96 Å). The resulting doped zirconia materials are termed stablized zirconias.[12]

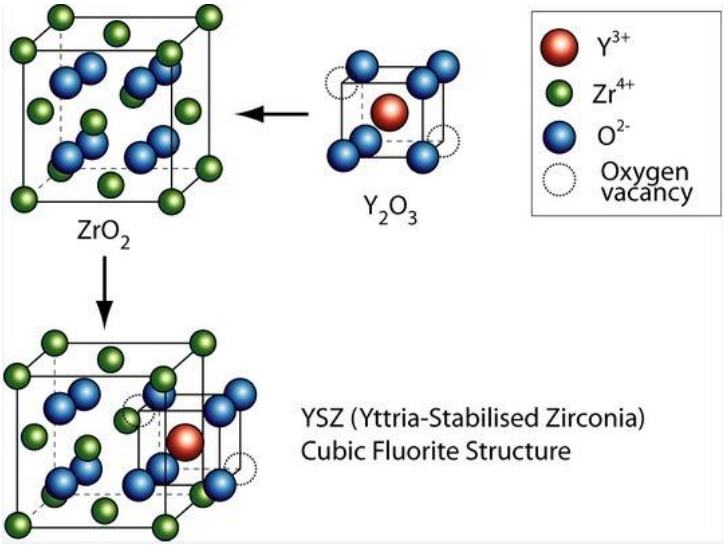


Fig 2.1: Yttria stabilized zirconia crystal lattice representation[12]

The addition of yttria to pure zirconia replaces some of the Zr^{4+} ions in the zirconia lattice with Y^{3+} ions. This produces oxygen vacancies, as three O^{2-} ions replace four O^{2-} ions. It also permits yttrium stabilized zirconia to conduct O^{2-} ions (and thus conduct an electrical current), provided there is sufficient vacancy site mobility, a property that increases with temperature. This ability to conduct O^{2-} ions makes yttria-stabilized zirconia well suited to use in solid oxide fuel cells, although it requires that they operate at high enough temperatures. The ionic conductivity of the stabilized zirconias increases with increasing dopant concentration (linearly for low dopant concentrations), then saturates, and then starts to decrease. The properties of interest to the engineer utilizing zirconia ceramics include strength, toughness, hardness, wear resistance and thermal properties.. The yttria content is the most significant controlling variable in yttria stabilized zirconia ceramics. In order to improve the mechanical properties it is essential to have a microstructure free of any monoclinic phase which would act as a flaw, and this dictates the minimum level of stabiliser added. Approximately 1.8 mol% yttria in solid solution results in a ceramic with a composition close to the phase boundary where the metastable tetragonal phase is

transformable. As a consequence high values of fracture toughness can be obtained with this composition. A maximum in the fracture strength is obtained at the 3 mol % yttria composition in research done so far. Tetragonal zirconia experiences a degradation when in contact with water at temperatures in the 200-300 ℃ range due to ageing of the metastable phase which restricts its use in long term applications [13].Biomedical grade zirconia exhibits the best mechanical properties of oxide ceramics. The mechanical properties of commercial yttria stabilized zirconia is given in Table 2.2[14].

Table 2.2: Mechanical properties of commercial yttria stabilized zirconia.

Mechanical properties of zirconia TZ-3Y	
Density	6.05 g/cm3
Hardness	1200 HV
Bend strength	900-1200 MPa
Compressive strength	2000 MPa
Fracture toughness	7-10 MPa/m1/2
Young's modulus	210 GPa

A Synthetic bone substitute should have similar strength to that of the cortical/cancellous bone being replaced (>200MPa). It should also have a similar modulus of elasticity to that of bone (20GPa) in an attempt to prevent both stress shielding and fatigue fracture under cyclic loading by maintaining adequate toughness [15].

CHAPTER 3

MATERIALS AND METHODS

EXPERIMENTAL PROCEDURE

CHARACTERISATION TECHNIQUES

HARDNESS TEST

BIOCOMPATIBILTY TEST

3.1 EXPERIMENTAL PROCEDURE

Commercial powders of titanium, zirconia and yttria and were used in synthesis of mechanically mixed composite samples. The samples were weighed in the electronic balance according to the following calculation:-

Mole% of Y_2O_3=3%

Mass fraction of zirconia=20% w/w

Total mass of composite to be prepared=5g

Molar mass of ytrria = 225.81g/mol

Molar mass of zirconia = 123.21g/mol

Thus, mass of zirconia = 5*0.02g =1g

Mass of yttria used = 0.03*225.81[mass of zirconia/123.21 + mass of yttria/225.81]

= 0.05g

Mass of titanium = (5-1-0.05)g = 3.95g.

3.1.1 BALL MILLING:-

The weighed samples were then ball milled in a SPEX 8000 mixer mill in inert gas atmosphere for 150 min and 300 min. The vial and the balls used in the milling procedure were made up of stainless steel. The ball to powder ratio was 10:1.

3.1.2 BINDER SOLUTION PREPARTION:-

4 %(w/v) PVA (Poly Vinyl Alchol) binder solution was prepared by 0.25 gm of PVA in 20 mL of distilled water. It was heated on a magnetic stirrer, simultaneously stirred. The weighed amount of PVA (0.25 gm) was added in small proportions, only when the previous addition had dissolved. It was ensured that the temperature remained below 90°C. Usually, constant heating is not required and it was heated in intervals but continuously stirred. Sometimes extra water was also added to keep up the required percentage of water. Continuous stirring resulted in a clear transparent solution. This binder was uniformly mixed with the ball milled sample in a mortar and kept to dry.

3.1.3 COMPACTION AND SINTERING:

After complete drying the hardened mass was finely ground.The dry ground powder was the measured to make the pellets. Green pellets of 7 mm diameter and 3 mm height were made by die pressing under a load of 2 tonnes (130 Mpa) with a dwell time of 20 sec. The pelletized samples were sintered at 1300 °C for 1 hr in normal atmosphere at 3^0C/min rise in temperature. This led to the oxidation of titanium to titanium oxide.

3.2 CHARACTERISATION TECHNIQUES:

3.2.1 SCANNING ELECTRON MICROSCOPE:

A scanning electron microscopy (SEM) is used for the check the surface morphology of the sample. In this type of electron microscope, images of the sample are scanned with the help electrons. The electrons interact with the atoms that generate the sample producing signals that contain information about the sample's surface composition, topography etc. At a potential of 15

KV, samples were scanned at different magnifications and images produced were used for further analysis.

3.2.2 X-RAY DIFFRACTION ANALYSIS

The composition of the titania-3Y PSZ composite samples was analyzed using X-Ray diffraction. Samples were studied in a X'PERT PANalytical X-Ray diffractometer with a graphite monochromator. X-Ray intensity was measured for angles in the range $20° < 2\Theta ° < 80°$ with scan rate of 3° per minute and time pulse of 1^0/min. The diffraction patterns produced were then compared with the existing data using JCPDS data file. To identify the phases present, the location of peaks in the XRD profiles were compared to reference spectra.

3.3 HARDNESS TEST:

The hardness measurement of the sintered composites was carried out with Vickers hardness tester. The model of the instrument used for finding out the Vickers hardness was LM248AT. Hardness of all the samples were measured under 300 gm loads.

3.4 BIOCOMPATIBILTY TEST (MTT ASSAY):

The cytotoxicity study was carried out for titania 3Y-PSZ sample with ball milling time for 150 and 300 min by direct contact method. MNCs from passage 4 were used for this study.The MTT assay has been performed following protocol given by Mosmann et al [16]. In brief, a cell suspension containing 5×10^4 cells was incubated in 96 well plates to get uniform monolayer. Then, sterilized composites were kept on the surface of monolayer in direct contact and incubated at 37^0C under 5% CO_2 for 24 hr. After incubation for 24 hr, composites were taken out from wells and media was removed. 10 µl of MTT solution (5 mg/ml) was added to respective well incubated at 37^0C for 4 hr. DMSO of 100 µl was used to solubilise formazan and absorbance was measured at 595 nm in a micro plate reader(Perkin Elmer 2030 explorer).

CHAPTER 4

RESULTS AND DISCUSSION

MORPHOLOGICAL CHARACTERIZATION

PHASE IDENTIFICATION

HARDNESS TEST

BIOCOMPATIBILITY STUDY

4.1. Morphological characterization

Figure 4.1 shows the scanning electron micrograph of the compacted titanium-3Y-PSZ composite after ball milling for two different time duration at different magnifications. Fig 4.1 (a) represents the ball milled titanium-3Y-PSZ composite for 150 mins at 500X magnification which shows fine grained particles forming agglomerates having nearly circular grain boundaries of non-uniform size while in case of fig 4.1(b) which represents ball milled titanium-3Y-PSZ composite for 300 mins at 500X magnification, the particle size is smaller compared to the earlier one. Cracks at the interfaces are also visible Further under higher magnification, in fig 4(c) the structure becomes more visible and uniform grain distribution with globular grain boundaries on the surface is visualised. Cracks can also be located at the interfaces. In fig 4(d) for the 300 min titanium-3Y-PSZ composite, the particle size has decreased significantly with slightly uniform grain size. The grains are globular with hexagonal boundaries. This indicates that with increase in ball mill duration the average particle size decreases and more agglomeration is formed among the particles.

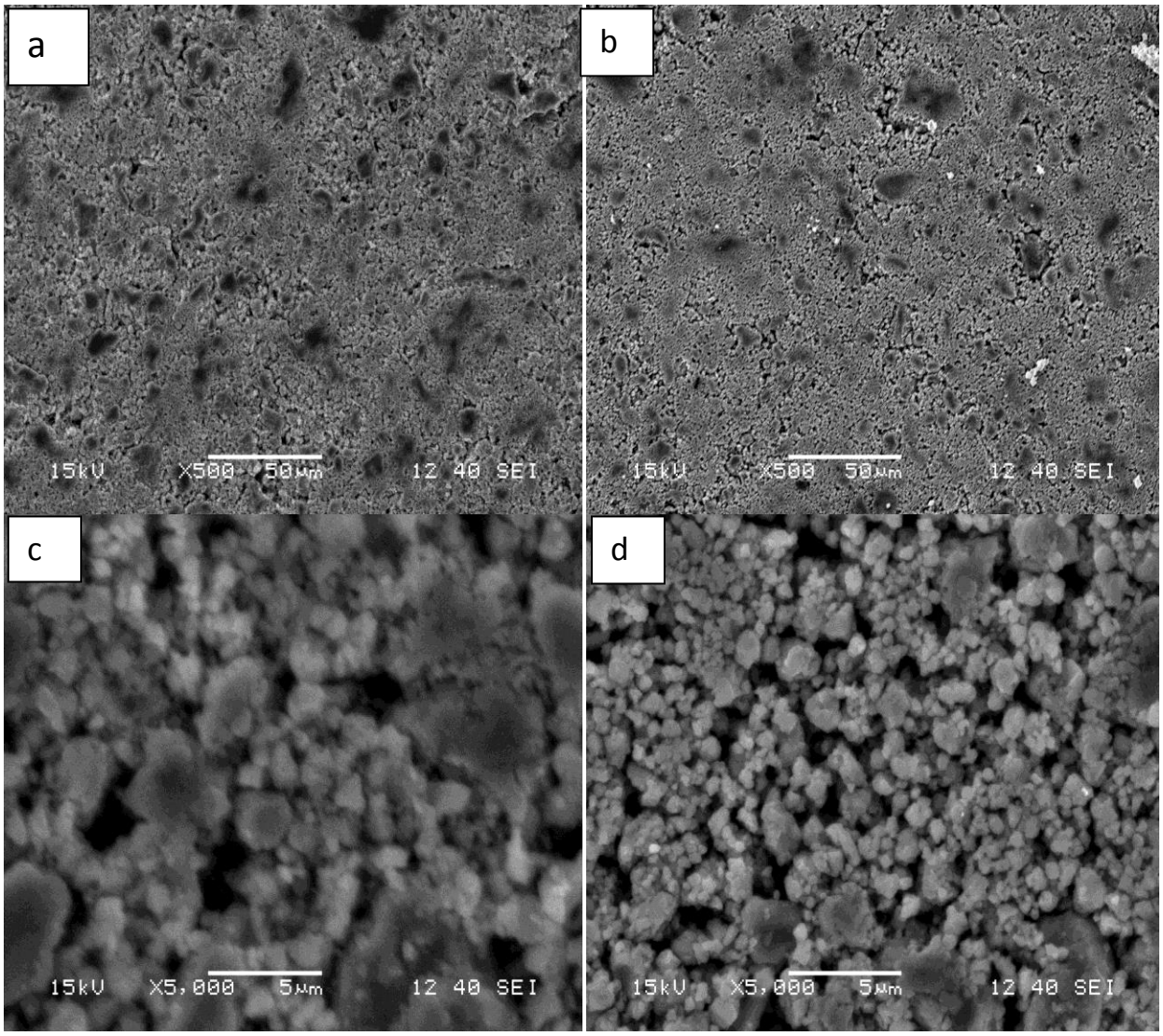
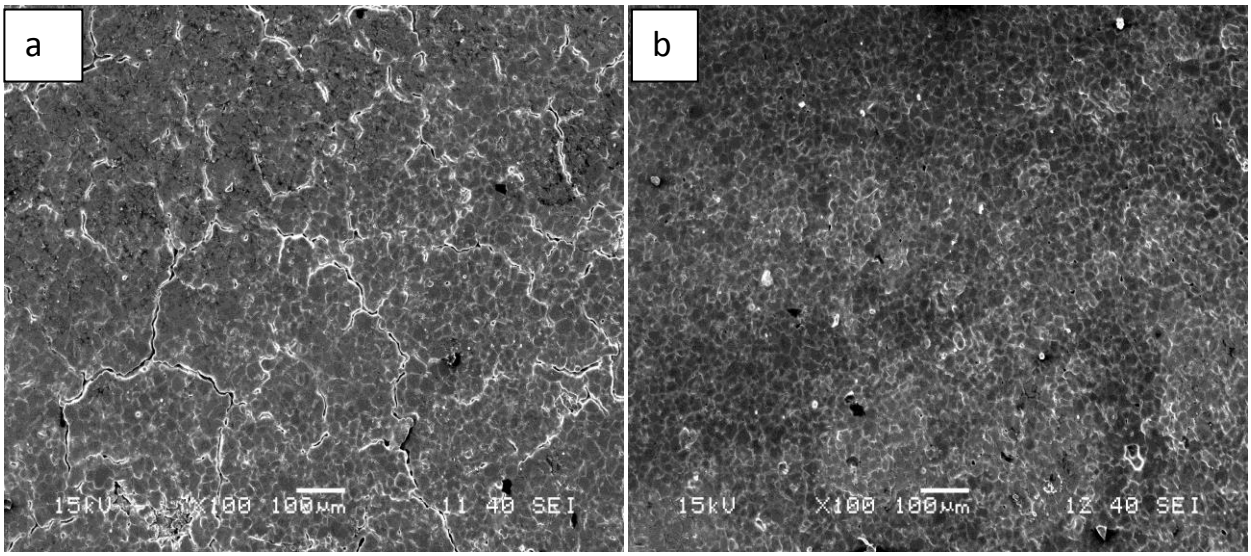


Figure 4.1*:* Scanning electron micrograph of compacted titanium-3Y-PSZ composite after ball milling for (a) 150 min at 500X (b) 300 min at 500 X (c) 150 min at 5,000X (d) 300 min at 5,000X

Figure 4.2 shows the scanning electron micrograph of the compacted titania-3Y-PSZ composite after sintering at 1300^{O} C for 1h at different magnifications. Fig 4.2(a) represents the 150 min titania-3Y-PSZ composite having nearly uniform grain size with some cracks at the grain interfaces while for the 300 min titania-3Y-PSZ composite in fig 4.2(b) the grains are more

compact and uniformly distributed. In fig 4.2(c) the sintered titania-3Y-PSZ composite of 150 min depict the formation of circular agglomerates which are more or less uniformly distributed. The particle size of this sample was calculated with the help of ImageJ software for 30 grains and the average grain size was found to be 23micrometers. In fig 4.2(d) the sintered titania-3Y-PSZ composite of 300 min exhibit more pore formation and non uniform grain size distribution having globular geometry. In this case the average grain size was calculated to be about 19.2 micrometers. Upon further magnifying the 150 min titania-3Y-PSZ composite in fig 4.2 (e) we observe that the grains are attached to each other at the interfaces which reveals that the grain boundary interaction between titania and zirconia composite are good enough. Fig 4.2(f) representing 300 min titania-3Y-PSZ composite also indicates the effective agglomeration at grain interface with hexagonal grain boundary.



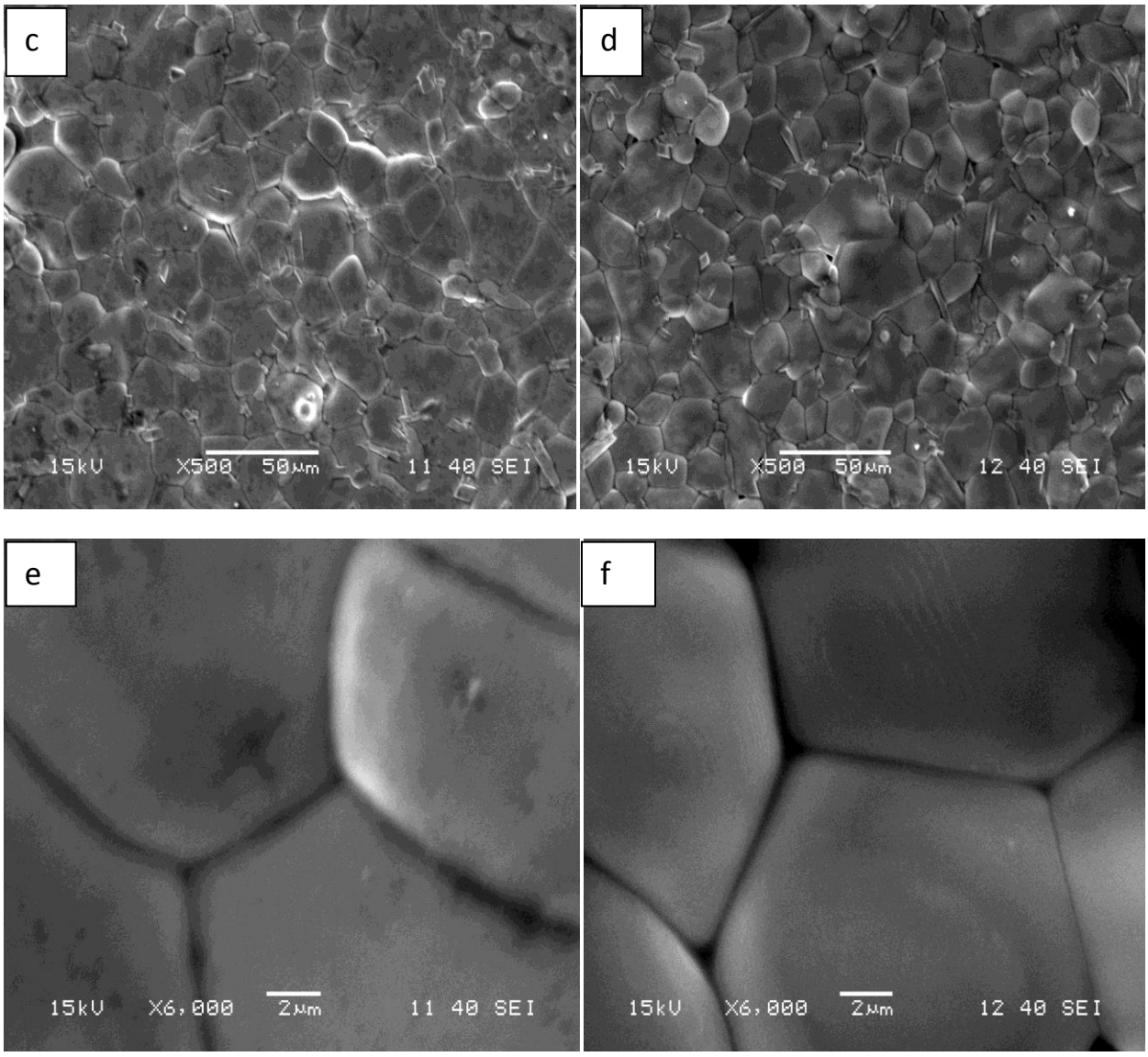


Figure 4.2*:* Scanning electron micrograph of the compacted titanium-3Y-PSZ composite after sintering at 1300^{O} C for 1h. (a) 150 min at 100X (b) 300 min at 100 X (c) 150 min at 500X (d) 300 min at 500X (e) 150 min at 6,000X (f) 300 min at 6,000 X

4.2. Phase identification:

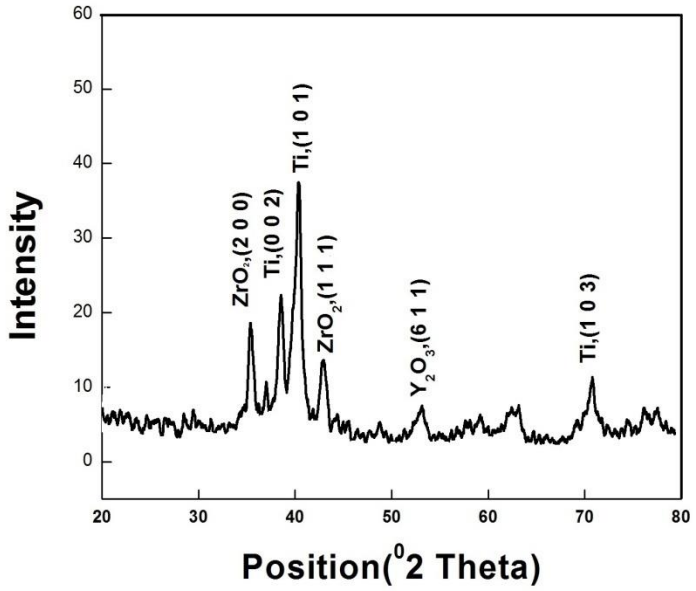
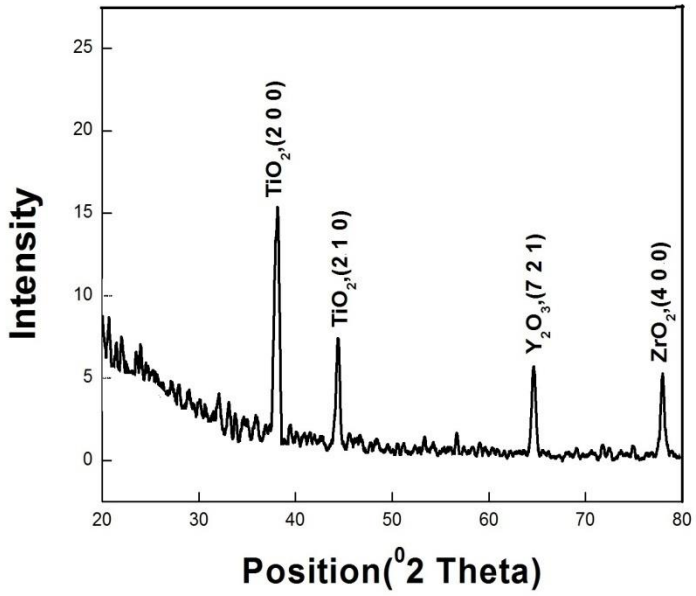
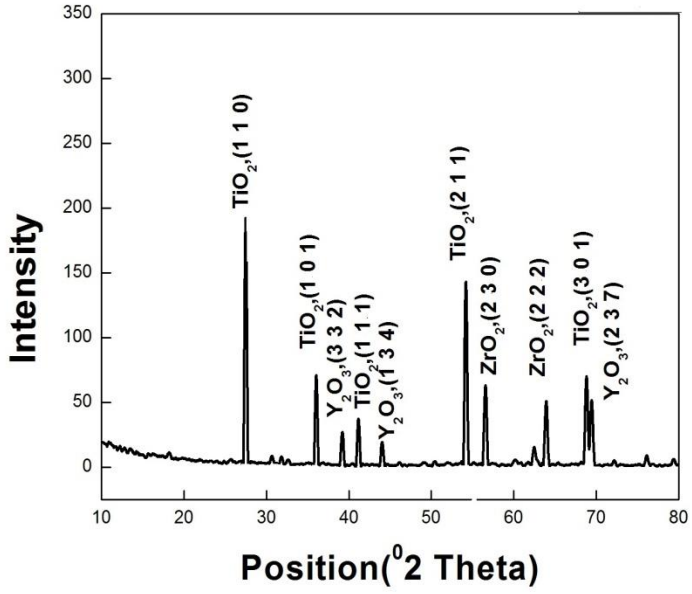


Figure 4.3 X-ray differaction plot of compacted titanium-3Y-PSZ composite after ball milling for 150 min.

The phase change before and after sintering was measured with XRD to study the monoclinic to tetragonal and titanium to titania phase change. Obtained peaks were matched with JCPDS data card no. 77-0442 (TiO_2), 27-0997 (ZrO_2), 76-0151 (Y_2O_3), 44-1294(Ti). The XRD patterns of compacted titanium-3Y-PSZ composite after ball milling for 150 min are represented in Fig. 4.3. The XRD patterns display the presence of titanium as the predominant phase along with a small amount of zirconia and yttria peaks. However, after the transformation of titanium into titania via sintering at 1300^{O} C for 1h, the intensity of the titania (TiO_2) increases with a concurrent increase in ZrO_2,Y_2O_3 peaks as in fig 4.4 . It can be deduced from fig 4.4 (b) more populated uniformly distributed peaks are obtained in case of the 300 min titania-3Y-PSZ.The presence of TiO_2 peaks confirm the oxidation of Ti into TiO_2 after sintering.



(a)



(b)

Figure 4.4 X-ray differaction plot of of the compacted titania-3Y-PSZ composite after sintering at 1300^{O}C for 1h after ball milling for (a) 150 min and (b) 300 min

4.3 Hardness test:

Table 4.3.1 Micro hardness value for sintered titania-yttria stabilized zirconia composite for 150 mins:

LOAD APPLIED(gm)	VICKER'S MICROHARDNESS(HV)
300	816.4
300	591.6
300	697.1
300	743.2
300	660.2

Table 4.3.2.Micro hardness value for sintered titania-yttria stabilized zirconia composite for 300 mins:

LOAD APPLIED(gm)	VICKER'S MICROHARDNESS(HV)
300	765.6
300	601.8
300	940.1
300	804.7
300	733.8

From the above tabulations of table 4.5.1 and 4.5.2 the average hardness values were calculated to be 769.2 HV and 701.7 HV for 300 min and 150 min sintered titania-3Y-PSZ. This illustrates that the sample having less particle size are more tough and are resistant to external load. This makes them potentially favourable for load bearing applications.

4.4 Biocompatibility study (MTT assay study) –

Fig 4.6.1 shows the biocompatibility study of sintered 300 min titania 3Y-PSZ sample. This reveals that the sample is biocompatible with better cell reproducibility on its surface.

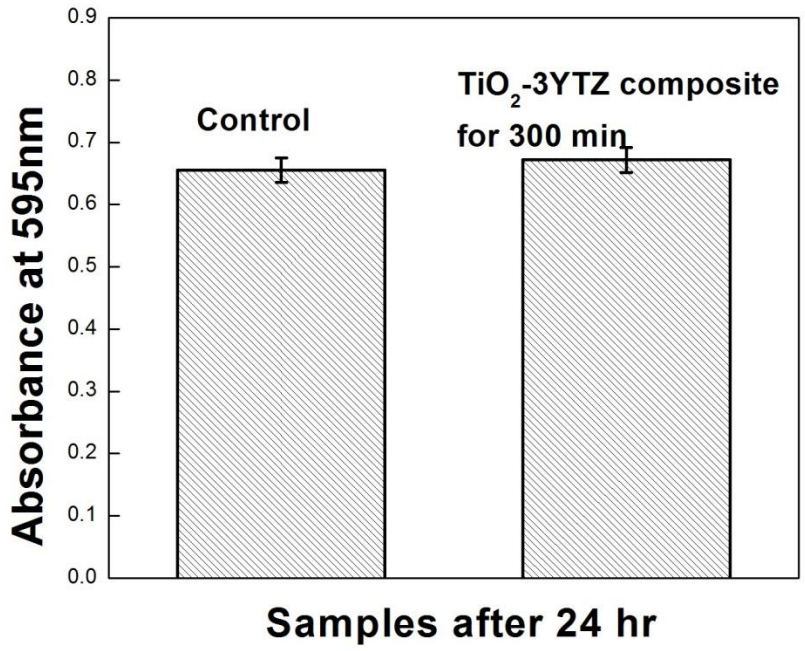


Fig.4.6.1 MTT assay test for cell toxicity and reproducibility for 300 min titania 3Y-PSZ sintered sample

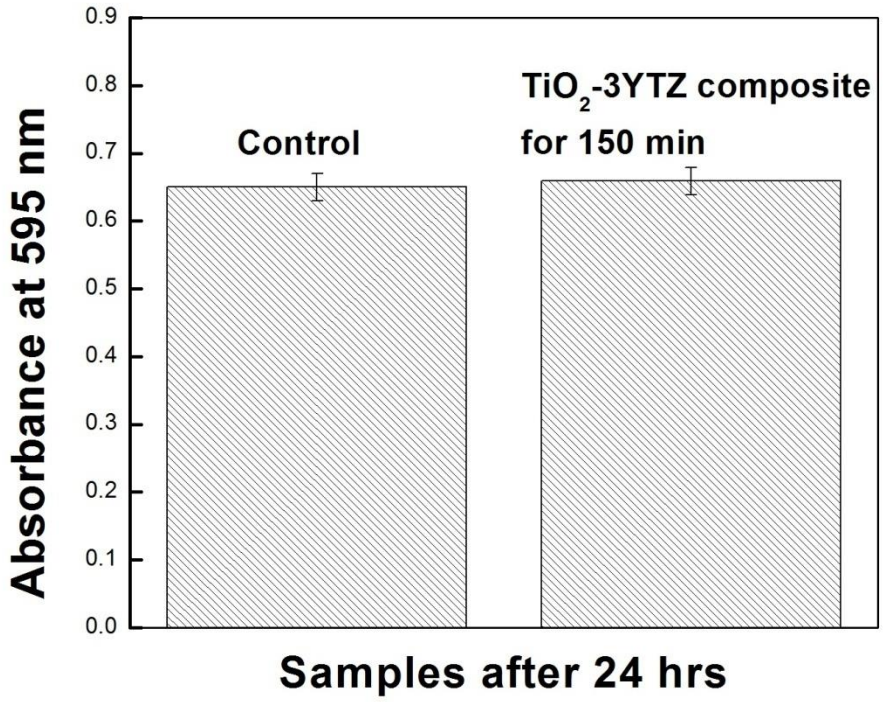


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Fig.4.6.2 MTT assay test for cell toxicity and reproducibility for 150 min titania 3Y-PSZ sintered sample.

The relationship between cell number and the amount of formazan generated was tested by measuring the intensity of absorbance. The absorbance was directly proportional to the number of cells in the culture medium (in approx.). From the Figure 4.6.1 it has been observed that the readings of the test samples are in good agreement with those of the control sample which shows there is rise in number of viable cells which represents the nontoxic and biocompatibility nature of titania 3Y-PSZ composite sample. The 300 min sample is likely to have a better cell growth as compared to that of the 150 min titania-3Y-PSZ composite.

CHAPTER 5

CONCLUSIONS

FUTURE WORK

REFERENCES

5.1 CONCLUSIONS

Synthesis of titania-yttria stabilized zirconia composite has been achieved following techniques like powder metallurgy, ball milling and subsequent sintering. The optimum concentrations of samples were determined[17]. The titania-yttria stabilized zirconia composite powders have been examined for optimization of particle size, particle morphology and phase analysis. The SEM studies reveal that the grain agglomerate with each other are quite well and is better in the 300 min ball milled composite. Hardness values are also impressive in order to withstand substantial amount of load. The phase analysis confirmed the presence of titania after conversion via sintering in normal atmosphere. The biocompatibility test revealed that the samples are biocompatible and do not for toxic compounds in the body environment and hence can be used as a reliable option in load bearing implants.

5.2 FUTURE WORK

It can be proposed that in the future research can be carried out in order to compare the results of metal-ceramic i.e titanium-3YPSZ and ceramic-ceramic titania-3YPSZ composite for application in load bearing implants. Characterisation techniques such as wear resistance and corrosion resistance can also be carried out.

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